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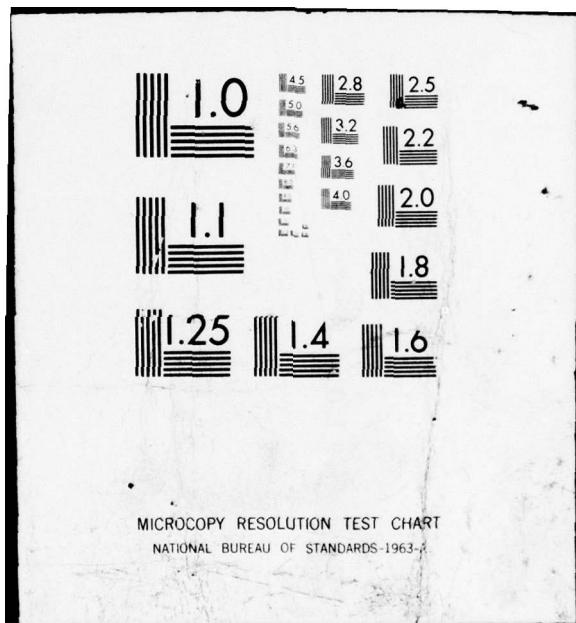
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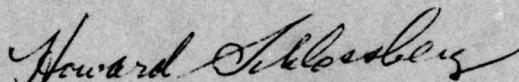
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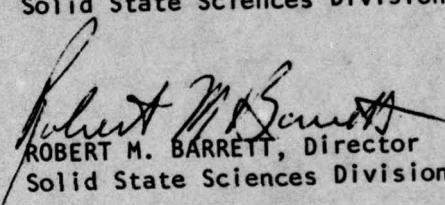
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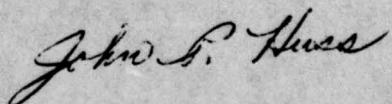
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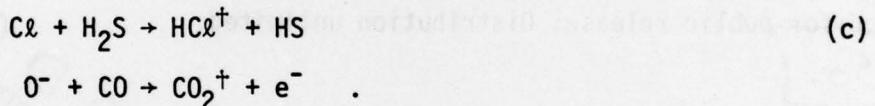
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20. Abstract (cont'd)

conditions. Of utmost significance has been our development of a new experimental method to observe for the first time infrared vibrational chemiluminescence from ion-molecule reactions. This technique offers promise for numerous detailed studies of the dynamics and infrared emission of ion-molecule reactions which are important to the Air Force.

The experimental program involves two distinct parts. The first employs a pulsed, tunable laser (purchased with this contract) to produce electronically excited atoms and other reactive atoms for studies of electronic-to-vibrational energy transfer and chemical reactions. Vibrational emission and chemiluminescence is observed with a sensitive infrared detector to measure the rates of energy transfer and to determine product state vibrational distributions. The second experimental system employs a flowing afterglow, normally used to study ion-molecule reactions, which was modified to observe ion-molecule vibrational chemiluminescence.

Systems studied include the reactions and energy transfer of:



Several important results have been obtained. Electronically excited  $\text{Br}^*$ -energy transfer to NO produces a population inversion between  $\text{NO}(V=2)$  and  $\text{NO}(V=1)$ . The electronically excited  $\text{Br}^*$  atom has been found to react substantially with  $\text{H}_2\text{S}$  to produce (vibrationally excited)  $\text{HBr}$ . The reaction of  $\text{Cl} + \text{H}_2\text{S}$  produces almost solely vibrational excitation of the  $\text{HCl}$  product. Finally, the  $\text{O}^- + \text{CO}$  associative detachment reaction was found to yield substantial excitation of the antisymmetric stretch in the  $\text{CO}_2$  product. The direct observation of vibrational chemiluminescence from the  $\text{O}^- + \text{CO}$  reaction is the first such demonstration of this kind. These results open up a whole new field of study for ion-molecule reactions.

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## FUNDAMENTAL KINETIC RATE PROCESSES OCCURRING IN POLYATOMIC VIBRATIONAL LASERS

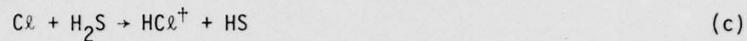
### 1. Report Summary

Laboratory experiments have been performed to study gaseous systems with regard to understanding electronic-to-vibrational energy transfer and chemical reactions of neutrals and ions which yield product state vibrational excitations. These results are important for the development of new methods to *pump infrared* vibrational lasers by energy transfer and by chemical reaction mechanisms. In addition, many chemical reaction systems are responsible for infrared emissions which are observed in the upper atmosphere under normal and perturbed conditions. Of utmost significance has been our development of a new experimental method to observe for the first time infrared vibrational chemiluminescence from ion-molecule reactions. This technique offers promise for numerous detailed studies of the dynamics and infrared emission of ion-molecule reactions which are important to the Air Force.

The experimental program involves two distinct parts. The first employs a pulsed, tunable laser (purchased with this contract) to produce electronically excited atoms and other reactive atoms for studies of electronic-to-vibrational energy transfer and chemical reactions. Vibrational emission and chemiluminescence is observed with a sensitive infrared detector to measure the rates of energy transfer and to determine product state vibrational distributions. The second experimental system employs a flowing afterglow, normally used to study

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## 2. Technical Results

Pulsed laser kinetic studies are carried out using a tunable CMX/4 flashlamp pumped dye laser, a large area InSb infrared detector, and a flow system. This work has been performed by Martin Braithwaite. The experimental arrangement is shown in figure 1. The flow cell with calibrated flow meters allows reliable study of reactive systems such as  $\text{Br}_2 + \text{NO}$ ,  $\text{Cl}_2 + \text{H}_2\text{S}$ ,  $\text{Br}_2 + \text{H}_2\text{S}$  and the like. Gases are mixed with injector inlets in a fast flow of argon carrier gas. Runs are made at varying flow rates to insure that no prereaction of the reagents has taken place.

The pulsed dye laser is crossed into the flow cell at right angles to the infrared detector port and the flow axis. The laser is typically multipassed with mirrors twenty times through the flow cell, to enhance the absorption by weak species. The laser source is used primarily for photodissociation of molecules such as  $\text{Br}_2$ ,  $\text{Cl}_2$  and  $\text{S}_2\text{Cl}_2$ , to generate ground and electronically excited atoms for these studies. All of the pulsed kinetic events are synchronized to the laser optical pulse with a photodiode trigger. The fluorescence

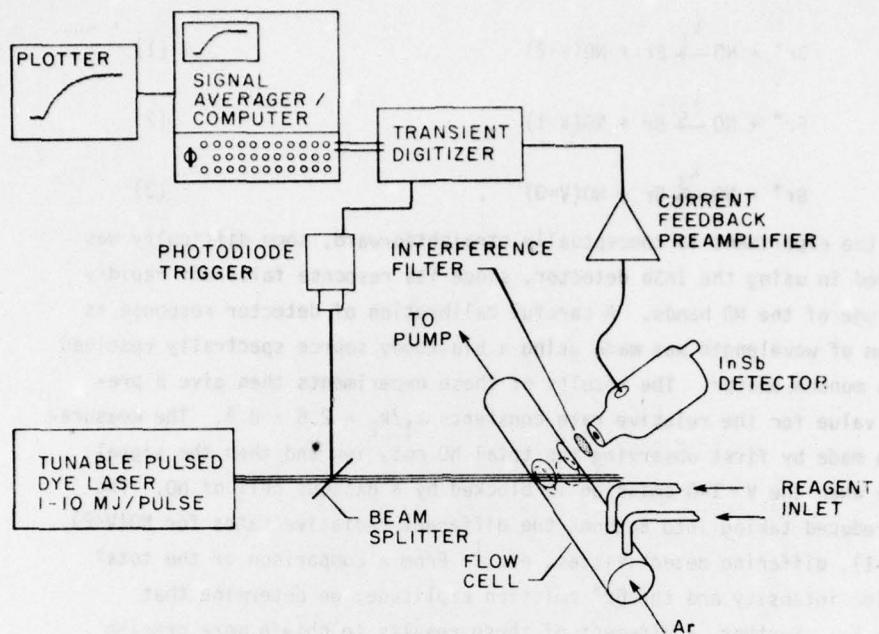


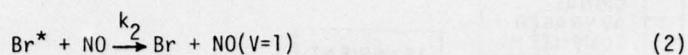
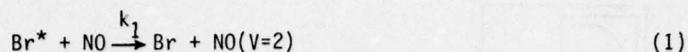
Figure 1. Experimental arrangement for pulsed laser kinetics using infrared fluorescence and chemiluminescence detection.

signals generated by the infrared detector are digitized with a Biomation 8100 transient recorder, and the digital information transferred to a signal averaging computer for enhancement. The resulting plots of emission intensity versus time have a typical signal-to-noise of 10-100. A variety of interference filters and gas blocking cells are used to isolate signals from the species of interest. In this manner both time and intensity information can be used to diagnose the rates and partitioning of the reaction energy.

(A)  $\text{Br}^* + \text{NO}$

The excitation energy of electronically excited  $\text{Br}(2p_{1/2})$  is  $3685 \text{ cm}^{-1}$ . This is sufficient to populate the nearly resonant  $V=2$  vibrational level of NO by E-V transfer. The E-V laser fluorescence technique is now well-established.<sup>1,2</sup> Such energy transfer processes are important for electronic-to-vibrational transfer lasers and the development of new molecular vibrational lasers in general.<sup>3,4</sup> Laser stimulated emission has been observed from  $\text{NO}(V=2 \rightarrow 1)$  upon E-V transfer from  $\text{Br}^*$ .<sup>2</sup>

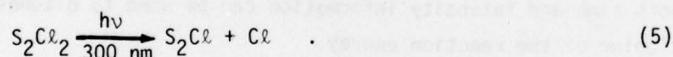
In these experiments, an accurate measurement of the branching of energy into  $\text{NO}(V=2)$  and  $\text{NO}(V=1)$  was attempted. The possible channels are:



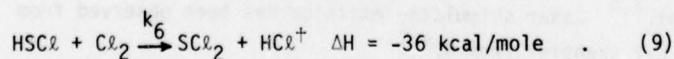
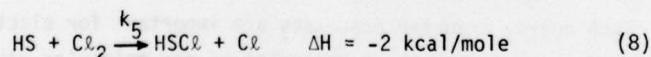
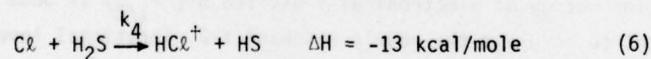
Although the experiment is conceptually straightforward, some difficulty was encountered in using the InSb detector, since its response falls off rapidly in the range of the NO bands. A careful calibration of detector response as a function of wavelength was made using a blackbody source spectrally resolved through a monochromator. The results of these experiments then give a preliminary value for the relative rate constants  $k_1/k_2 = 2.6 \pm 0.5$ . The measurements are made by first observing the total NO emission and then the signal amplitude when the  $\text{V}=1-0$  emission is blocked by a gaseous cell of NO. The data is reduced taking into account the different radiative rates for  $\text{NO}(\text{V}=2)$  and  $\text{NO}(\text{V}=1)$ , differing detectivities, etc.<sup>1</sup> From a comparison of the total NO emission intensity and the  $\text{Br}^*$  emission amplitude, we determine that  $k_1 + k_2 > k_3$ . Further, refinement of these results to obtain more precise quantitative data is presently being carried out.

(B)  $\text{Cl} + \text{H}_2\text{S}$

The reaction system of Cl atom with  $\text{H}_2\text{S}$  was studied to determine the extent of vibrational excitation in each of the product molecules. Two sources of Cl atom by photolysis were used:



In the case of photolysis of  $\text{Cl}_2$  in the flow of  $\text{H}_2\text{S}$ , the following reactions appear to be important:



The experimental results demonstrate conclusively that the reaction system

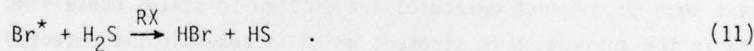
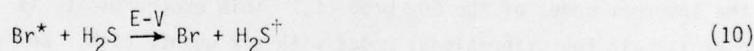
proceeds via the chain (7) and (8). This was determined from the slow but continuously rising emission signal of the vibrationally excited HCl product. The amplitude of the observed signal was ten times greater than the signal obtained when a similar density of Cl atoms is generated in a flow of HBr, a reaction which has no possibility of chain steps.

Using the photolysis of  $S_2Cl_2$  as a source of Cl atoms was found to inhibit the chain step (8), and the rise time of the HCl emission from reaction (7) could be studied independently. Extensive measurements of the  $Cl + H_2S$  reaction rate constant gives  $k_4 = 6.2 \pm 0.5 \times 10^{-11} \text{ cm}^3 \text{ molecule}^{-1} \text{ sec}^{-1}$ . Analysis of the emission from reaction (7) shows that HCl is formed only in the ( $V=1$ ) state. This is consistent with the available reaction exothermicity. No detectable emission is observed from the HS fragment. An independent and highly reliable calculation of the relative transition strengths for spontaneous radiative emission of HCl and HS gives  $\tau_{HCl}/\tau_{HS} = 0.10$ .<sup>5</sup> Thus we are able to assign a limit to the extent of vibrational excitation in the HS fragment of <10%. This is in agreement with the qualitative model for reacting A + BC systems which would say that the reaction exothermicity is deposited in the newly formed band when the activation barrier comes early in the entrance channel of the potential surface.

Further study of the chain reaction system with  $Cl_2$  as the photolysis source showed that half of the HCl is also produced in excited states greater than  $V=1$ . This can only be explained by some reaction other than (7). Reaction (9) is one which could possibly excite HCl to higher vibrational levels because of its large exothermicity. If this reaction does go efficiently, a very unique rearrangement must be occurring, which will be important to study further.

(C)  $Br^* + H_2S$

A further study of E-V transfer events in reactive systems involves the interaction of  $Br^*$  with  $H_2S$ . In this case, a competition between E-V transfer and a reactive channel is possible:



In addition, the reactive channel (11) has the possibility of exciting either the HBr or HS to  $V=1$ .

The dye laser was tuned to 580 nm, where photolysis of  $\text{Br}_2$  produces only ground state Br atoms. As expected from the exothermicity of the  $\text{Br} + \text{H}_2\text{S}$  reaction, no vibrationally excited species were observed in the products. On tuning the dye laser to 480 nm, where photolysis of  $\text{Br}_2$  produces one  $\text{Br}^*$  and one Br atom, vibrationally excited product emission was observed. Insertion of a gas filter cell of HBr blocked the emission signal totally, indicating that the  $\text{Br}^*$  reacts with  $\text{H}_2\text{S}$  to produce  $\text{HBr}(V=1)$ . No emission was observed from vibrationally excited  $\text{H}_2\text{S}$  down to the limit of detectability. Thus it appears that reaction (11) and not E-V transfer (10) is the dominant process. Similar to the reaction of Cl with  $\text{H}_2\text{S}$ , no vibrationally excited HS was observed either. Preliminary intensity data for the  $\text{HBr}(V=1)/\text{Br}^*$  ratio indicates that the reactive channel is a substantial fraction of the total quenching rate of  $\text{Br}^*$  by  $\text{H}_2\text{S}$ .

(D)  $\text{O}^- + \text{CO}$ : ion-molecule vibrational chemiluminescence

Further emphasis to study reactive dynamics of relevance to the interests of the Air Force has led our group to develop a completely new technique to study ion-molecule reactions. This work was performed by Veronica M. Bierbaum (research associate with C. H. DePuy), G. Barney Ellison (research associate with W. C. Lineberger), Jean H. Futrell (JILA Visiting Fellow), and the principal investigator. The work is important because ion-molecule reactions are responsible for numerous infrared emissions observed in the upper atmosphere. Our technique allows a direct observation of such chemiluminescence emissions from ion-molecule reactions, and provides a method to study the dynamics of ion-molecule reactions.

The  $\text{O}^- + \text{CO}$  associative detachment reaction was chosen because it has a minimum of obscuring effects and because the  $\text{CO}_2$  product is easily observed in emission at 4.3  $\mu\text{m}$ . The thermal rate constant is  $6.5 \times 10^{-10} \text{ cm}^3 \text{ molecule}^{-1} \text{ sec}^{-1}$ .<sup>6</sup> A substantial fraction of the electrons are ejected with low translational energy, suggesting that most of the 4.0 eV reaction exothermicity is taken up in the internal modes of the  $\text{CO}_2$  product.<sup>7</sup> This exothermicity is sufficient to excite all four vibrational modes with six quanta each. We may assume at least some  $\text{CO}_2$  product molecules are excited to states containing one or more quanta in the antisymmetric stretch, which is conveniently detected at 4.3  $\mu\text{m}$  with available detectors.

The flowing afterglow (figure 2), used successfully for many years to measure rate constants of ion-molecule reactions, offers a carefully controlled

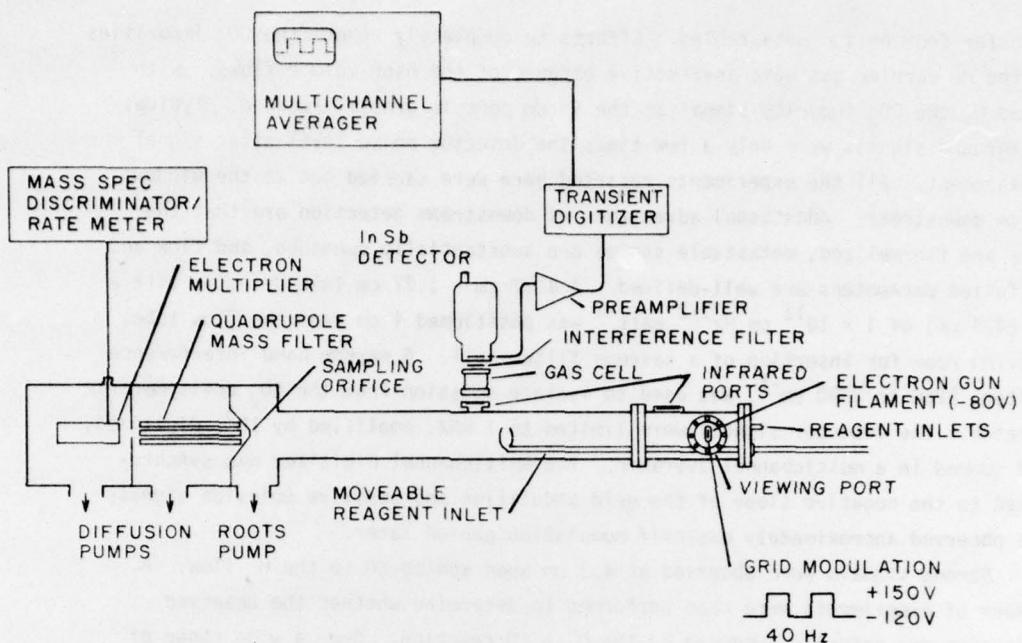


Figure 2. Experimental apparatus for observing ion-molecule vibrational chemiluminescence in a flowing afterglow.

source of positive and negative ions.<sup>8</sup>  $O^-$  ions are produced in the ion source by dissociative electron attachment of  $O_2$  with electrons. Typical conditions are 0.5 torr He, 0-30 mtorr  $O_2$ , and emission currents of 0.2-50 ma. The filament potential is maintained at -80 V, and a fine mesh tungsten grid in front of the filament is modulated with a square wave from -120 V to +150 V at a frequency of 40 Hz. Under these conditions, 100% modulation of the electrons is obtained and phase sensitive detection of the optical signal is optimized.

Ion densities are estimated from the signal detected with a quadrupole mass spectrometer located 125 cm downstream from the ion source. Both the positive and negative ion mass spectra were very clean, consisting primarily of  $O^-$ ,  $O_2^-$ ,  $O^+$ ,  $O_2^+$  and some impurity  $NO_2^-$  and  $Cl^-$ . Of these ions, only  $O^-$  is potentially reactive with CO. Densities of  $O^-$  55 cm from the ion source were estimated to range from  $10^6$  -  $10^8$ /cc.

Infrared emission was observed at two port windows, 10 cm and 55 cm from the ion source. At the 10 cm port, strong emission was always observed from low level  $CO_2$  impurities in the helium plasma, presumably excited by energy

transfer from helium metastables. Efforts to completely remove the  $\text{CO}_2$  impurities in the He carrier gas were ineffective because of the high volume flows. With added  $\text{O}_2$  the  $\text{CO}_2$  impurity signal at the 55 cm port is greatly reduced. Typical background signals were only a few times the detector noise level after signal enhancement. All the experiments reported here were carried out at the window 55 cm downstream. Additional advantages of downstream detection are that the ions are thermalized, metastable states are substantially quenched, and flow and diffusion parameters are well-defined. A  $1.27 \text{ cm} \times 1.27 \text{ cm}$  InSb detector with a  $D^*$  (4.3  $\mu\text{m}$ ) of  $1 \times 10^{11} \text{ cm Hz}^{1/2} \text{ watt}^{-1}$  was positioned 4 cm from the flow tube, leaving room for insertion of a gaseous filter cell. A narrow band interference filter, 2250 to 2390  $\text{cm}^{-1}$ , was used to isolate emission from the  $\text{CO}_2$  antisymmetric stretch. The detector signals were limited to 1 kHz, amplified by  $10^5$ , digitized, and summed in a multichannel averager. The multichannel digitizer was synchronized to the negative slope of the grid modulation and positive emission signals are observed approximately one-half modulation period later.

Strong signals were observed at 4.3  $\mu\text{m}$  upon adding CO to the  $\text{O}^-$  flow. A number of experiments were then performed to determine whether the observed emissions are actually produced by the  $\text{O}^- + \text{CO}$  reaction. Over a wide range of  $\text{O}_2$  and He pressures and emission current conditions, the background emission signals from  $\text{CO}_2$  impurities in the He and CO were found to be a factor of ten less than the signals from the  $\text{O}^- + \text{CO}$  reaction. When the  $\text{O}^-$  density was varied by both changes in emission current and added  $\text{O}_2$ , the  $\text{CO}_2$  emission intensity was found to be directly correlated with the  $\text{O}^-$  concentration. The  $\text{CO}_2$  emission intensity also followed the expected dependence on added CO. CO pressures of 0.10 mtorr reacted completely with available  $\text{O}^-$  over a distance of 20 cm, and the  $\text{CO}_2$  emission intensity did not increase on adding up to 1 mtorr additional CO. A  $\text{CO}_2$  gas filter cell, 1 cm and 1 atm, was used to block emission only from  $(001) \rightarrow (000)$ . Measurements of signals from plasma excited  $\text{CO}_2$  impurity molecules gave a 5% transmission through the gas cell. This corresponds approximately to the Boltzmann ratio of (011) to (001) at room temperature. When the same experiment was performed on the product molecules of the  $\text{O}^- + \text{CO}$  reaction, 30% transmission was observed, indicating a distinctly nonBoltzmann distribution.

We conclude from these experiments that emission from the  $\text{CO}_2$  antisymmetric stretch is observed in the product of  $\text{O}^- + \text{CO}$  reaction. We must consider whether this vibrational mode is excited directly or by some energy transfer mechanism. The density of unquenched helium metastables at the CO reagent inlet is far too low to produce the magnitudes of the signals observed. In the gas filter

experiments, the CO inlet was only 1 cm from the viewing port, corresponding to about  $10^3$  collisions of the  $\text{CO}_2$  product predominantly with helium while it is observed. From the known relaxation rate of  $\text{CO}_2(001)$  with helium, more than  $10^5$  collisions are required for intramolecular energy transfer.<sup>10</sup> Unless the intramolecular relaxation rates for more highly excited  $\text{CO}_2$  molecules are  $10^2$  times faster than for the (001) state, it is unlikely that the antisymmetric stretch is populated from other modes by a subsequent intramolecular energy transfer mechanism. We conclude that some of the  $\text{CO}_2$  is excited directly with one or more quanta in the antisymmetric stretch.

A qualitative description of the dynamics of the associative detachment process has been given.<sup>9</sup> Since the ground state of  $\text{CO}_2^-$  is bent, and that of  $\text{CO}_2$  is linear, the  $\text{CO}_2$  is expected to receive multiple quanta in the bending vibration in the autodetachment process. Oscillations are observed in the energies of the electrons ejected from the  $\text{O}^- + \text{CO}$  reaction which suggests excitation of a progression in the bending mode series.<sup>7</sup> This is in contrast to the observed progressions in the symmetric stretch series for electron excitation of  $\text{CO}_2$  in the 4 eV range.<sup>10</sup> This process most likely samples a different part of the  $\text{CO}_2^-$  compound state surface. The details of the vibrational excitation depend in each case on the  $\text{CO}_2^-$  lifetime and the exact nature of the potential surface crossing. The present experiments demonstrate that at least one quantum of excitation is populated in the antisymmetric stretch for some of the product molecules of the  $\text{O}^- + \text{CO}$  reaction. This result is consistent with the picture that all the vibrational modes should be excited to some extent,<sup>7,11</sup> with perhaps substantial excitation in the bending mode. Further experiments with a range of detector wavelengths will be required to describe the distribution of vibrational excitation quantitatively.

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## METRIC SYSTEM

### BASE UNITS:

Quantity	Unit	SI Symbol	Formula
length	metre	m	...
mass	kilogram	kg	...
time	second	s	...
electric current	ampere	A	...
thermodynamic temperature	kelvin	K	...
amount of substance	mole	mol	...
luminous intensity	candela	cd	...

### SUPPLEMENTARY UNITS:

plane angle	radian	rad	...
solid angle	steradian	sr	...

### DERIVED UNITS:

Acceleration	metre per second squared	...	m/s
activity (of a radioactive source)	disintegration per second	...	(disintegration)/s
angular acceleration	radian per second squared	...	rad/s
angular velocity	radian per second	...	rad/s
area	square metre	...	m <sup>2</sup>
density	kilogram per cubic metre	...	kg/m <sup>3</sup>
electric capacitance	farad	F	A·s/V
electrical conductance	siemens	S	A/V
electric field strength	volt per metre	...	V/m
electric inductance	henry	H	V·s/A
electric potential difference	volt	V	V/A
electric resistance	ohm	Ω	W/A
electromotive force	volt	V	W/A
energy	joule	J	N·m
entropy	joule per kelvin	...	J/K
force	newton	N	kg·m/s
frequency	hertz	Hz	(cycle)/s
illuminance	lux	lx	lm/m <sup>2</sup>
luminance	candela per square metre	...	cd/m <sup>2</sup>
luminous flux	lumen	lm	cd·sr
magnetic field strength	ampere per metre	...	A/m
magnetic flux	weber	Wb	V·s
magnetic flux density	tesla	T	Wb/m
magnetomotive force	ampere	A	...
power	watt	W	J/s
pressure	pascal	Pa	N/m
quantity of electricity	coulomb	C	A·s
quantity of heat	joule	J	N·m
radiant intensity	watt per steradian	...	W/sr
specific heat	joule per kilogram-kelvin	...	J/kg·K
stress	pascal	Pa	N/m
thermal conductivity	watt per metre-kelvin	...	W/m·K
velocity	metre per second	...	m/s
viscosity, dynamic	pascal-second	...	Pa·s
viscosity, kinematic	square metre per second	...	m <sup>2</sup> /s
voltage	volt	V	W/A
volume	cubic metre	...	m <sup>3</sup>
wavenumber	reciprocal metre	...	(wave)/m
work	joule	J	N·m

### SI PREFIXES:

Multiplication Factors	Prefix	SI Symbol
$1\ 000\ 000\ 000\ 000 = 10^{12}$	tera	T
$1\ 000\ 000\ 000 = 10^9$	giga	G
$1\ 000\ 000 = 10^6$	mega	M
$1\ 000 = 10^3$	kilo	k
$100 = 10^2$	hecto*	h
$10 = 10^1$	deka*	da
$0.1 = 10^{-1}$	deci*	d
$0.01 = 10^{-2}$	centi*	c
$0.001 = 10^{-3}$	milli	m
$0.000\ 001 = 10^{-6}$	micro	μ
$0.000\ 000\ 001 = 10^{-9}$	nano	n
$0.000\ 000\ 000\ 001 = 10^{-12}$	pico	p
$0.000\ 000\ 000\ 000\ 001 = 10^{-15}$	femto	f
$0.000\ 000\ 000\ 000\ 000\ 001 = 10^{-18}$	atto	a

\* To be avoided where possible.